

Application

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Extraction of Phthalates and Adipates from Water, Using Solid Phase Extraction Disks

ENVI-Disk solid phase extraction disks are a porous glass fiber matrix containing C8- or C18-modified silica. In extractions of organic contaminants from 1 liter or more of water, these rigid disks provide faster flow rates and exhibit less clogging than Teflon® disks, and are less expensive. ENVI Disks can be used to extract polynuclear (polycyclic) aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, herbicides, and phthalates from water. This application note describes a process for extracting phthalates and adipates from water according to US Environmental Protection Agency Method 506.

Key Words:

- semivolatiles • phthalates • adipates • water analyses
- SPE disks • sample preparation

NOTE: This method is based on the sample extraction procedure in Section 11.3 of US Environmental Protection Agency Method 506. It is not intended to be a replacement or substitute for the EPA procedure. For detailed information about preparing samples for analysis according to Method 506, please refer to the EPA method. (Request from National Technical Information Service, U.S. Department of Commerce, Springfield, VA 22161 USA [Tel. 703-487-4650])

Sample Preparation

1. Allow a 1 liter sample of water to equilibrate to room temperature in a narrow mouth amber glass bottle.
2. Add 5mL methanol and mix thoroughly.
3. For QC/QA samples, add appropriate levels of each analyte.

Apparatus Assembly

1. Assemble the 47mm glassware (flask, vacuum line, and filtration support) according to the instructions that accompanied the ENVI-Disk™ SPE disks.
2. Place a 47mm ENVI™-18 DSK disk (Cat. No. 57171) on the support, **wrinkled side up**.
3. Center the disk, so that it evenly overlaps all sides of the support.
4. Carefully place the reservoir on the disk and attach the clamp securely.

Disk Cleaning

1. Pour or pipette 5mL methylene chloride onto the disk and immediately draw the liquid through the disk under moderate vacuum (15" Hg/50kPa). Maintain vacuum for 5 minutes, to remove all solvent.

Disk Conditioning

1. Pour or pipette 5mL methanol onto the disk and immediately apply low vacuum (1–2" Hg/3–7kPa). Release the vacuum when the methanol is just above the top surface of the disk. **Do not allow any air to pass through the disk or to reach the top surface of the disk.**
2. Immediately pour or pipette 5mL deionized water onto the disk and immediately apply low vacuum. Release the vacuum when the water is just above the top surface of the disk.
Note: In steps 1 and 2 it is better to leave extra liquid covering the disk than to allow air to contact the surface of the disk.

Sample Addition

1. Pour the sample into the apparatus, directly onto the film of water left on the disk from the last conditioning step. Invert the sample bottle in the reservoir and allow the sample to automatically feed onto the disk.
2. Adjust the vacuum to approximately 10" Hg (35kPa), to provide a flow rate of approximately 100mL/minute. The disk must not go dry until the entire sample has been processed.

Analyte Elution

1. Release the vacuum.
2. Remove the filtration support and reservoir from the vacuum flask without disturbing the disk.
3. Empty the processed water from the flask, insert the sample collection tube, and reassemble the apparatus.
4. Add 5mL acetonitrile to the sample bottle, cap the bottle, and gently swirl it to rinse all of the inside surface. Allow the bottle to stand for 1–2 minutes.
5. Transfer the acetonitrile to the disk, using a glass pipette and rinsing the sides of the reservoir in the process.
6. Draw the solvent through the disk (5" Hg/17kPa vacuum).
7. Repeat steps 4–6 with fresh acetonitrile, then repeat steps 4–6 twice with methylene chloride, combining all eluates in the sample collection tube.

Analysis

1. Remove any remaining water from the eluate by passing it through approximately 3g anhydrous sodium sulfate.
2. Concentrate the eluate to 1mL and analyze 1–2µL by GC/PID or GC/MS, following the procedure in EPA Method 506.



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Ordering Information:

Description	Cat. No.
ENVI-18 DSK SPE Disks	
C18 bonded phase	
47mm, pk. of 24	57171
90mm, pk. of 12	57170-U
PTE™-5 Capillary GC Column	
30m x 0.25mm ID, 0.25µm film	24135-U

For filtration apparatus to use with ENVI Disk SPE disks, refer to the current Supelco catalog or call your Supelco representative.

References

1. US Environmental Protection Agency Method 506.
Request from National Technical Information Service, U.S. Department of Commerce, Springfield, VA 22161 USA (Tel. 703-487-4650).

Trademarks

ENVI, ENVI-Disk, PTE – Sigma-Aldrich Co.
Teflon – E.I. du Pont de Nemours & Co., Inc.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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